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RESEARCH MEMORANDUM

STATIC CRUCIBLE INVESTIGATION OF CORROSION AND
MASS-TRANSFER OF NICKEL IN MOLTEN SODIUM
HYDROXIDE WITH VARIOUS ADDITIVES

By Americo F. Forestieri and William F. Zelezny

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

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RESEARCH MEMORANDUMSTATIC CRUCIBLE INVESTIGATION OF CORROSION AND MASS-TRANSFER
OF NICKEL IN MOLTEN SODIUM HYDROXIDE WITH VARIOUS ADDITIVES

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SUMMARY

The effects of a number of additives on the phenomena of corrosion and mass transfer of nickel by molten sodium hydroxide in the neighborhood of 1500° F have been investigated by means of static crucible tests. The experimental conditions maintained in order to obtain a satisfactory degree of reproducibility are explained in detail. The additives used are classified as to their effects: detrimental, inert, and beneficial. Additives found to reduce mass transfer in this investigation were: calcium hydride, sodium aluminate, and hydrogen.

INTRODUCTION

One of the major problems in the practical utilization of nuclear energy for the propulsion of aircraft is the transfer of the energy, released as heat at relatively high temperature levels within the nuclear reactor, to propulsion devices such as turbojet engines. The solution of this problem requires the development or discovery of a suitable heat-transfer fluid, together with a material which will contain this fluid without suffering excessive attack chemically or otherwise. Molten sodium hydroxide is in many respects an attractive choice for this fluid because of its favorable liquid range, excellent heat-transfer characteristics, moderating properties, and low capture cross section. On the other hand, molten sodium hydroxide has produced mass transfer in all metals or alloys which have been used in attempts to contain it and has also proven exceedingly corrosive, in the usual chemical sense, to most of these metals and alloys.

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In preliminary experiments in static capsules, difficulty was experienced in obtaining reproducible mass-transfer and corrosion results. Accordingly, small trends, representing the effects of some additive for example, were lost in the scatter of the data. Since these trends are important in the establishment of the direction of further work, it was deemed important to set up an experimental procedure of sufficient reproducibility to enable small effects to be determined accurately. Because this required very accurate control of the conditions that materially affect the results, a study was therefore undertaken prior to the experiments with additives to determine the factors that are important in affecting the accuracy and reproducibility of corrosion tests. The experimental procedures developed in this study were used in the investigation conducted at the NACA Lewis laboratory to determine the effects of additives.

Among the variables taken into consideration were the following: uniformity of composition of materials used in the various tests, surface conditions (especially cleanliness) of the crucibles and specimens, prevention of contamination of the sodium hydroxide with atmospheric moisture and carbon dioxide, maintenance of uniform moisture content of the sodium hydroxide, filling of the crucibles to a uniform height with sodium hydroxide, and the maintaining of similar and reproducible temperature conditions in all crucibles. Of these experimental variables the last two proved to be especially important. Sufficient attention to these details, as described in the section on apparatus and procedure, was successful in achieving a degree of reproducibility which was deemed satisfactory for the purposes of this investigation.

Of all the metals tested to date, nickel shows the least corrosion by molten sodium hydroxide, although the phenomenon of mass transfer is evident to a marked degree. Regardless of this undesirable property, nickel still appears to be the best metal for the purpose of containing molten sodium hydroxide. Commercial pure nickel, which for practical purposes may be regarded as consisting of a single phase, also offers the advantage of providing a system which is simple in comparison with many alloys in which several phases may be present. This simplicity of the system facilitates the evaluation of experimental results. Accordingly, nickel was the metal chosen for these studies.

Because nickel is subject to mass-transfer by molten sodium hydroxide, an experimental program was therefore initiated to investigate the effect of a series of additives on the corrosion and mass-transfer of nickel by sodium hydroxide at 1500° F. These experiments were made in nickel crucibles in which a nickel specimen was located at the bottom of the crucible with the sodium hydroxide and additives, if any, filling approximately 50 percent of the volume of the crucible. A temperature difference was maintained between the bottom and top of the crucible, the bottom being hotter than the top to induce circulation by

convection; the rate of mass transfer was thereby increased. These experiments were static in the sense that no forced circulation of the molten sodium hydroxide, other than the thermal convection just mentioned, took place. The occurrence of mass transfer made itself evident both by the loss in weight of the nickel specimen at the bottom of the crucible and by the deposition of crystals of nickel at the upper surface of the sodium hydroxide. The following additives were investigated: sodium carbonate, palladium, nickel oxide, hydrated sodium chromate, chromium oxide, sodium chlorate, sodium oxide, sodium hydride, lithium hydride, sodium chloride, calcium nitride, calcium hydride, sodium aluminate, and hydrogen.

APPARATUS AND PROCEDURE

General outline of procedure. - The procedure followed in this investigation will be outlined briefly here and then discussed more fully in subsequent paragraphs. Nickel capsules or crucibles as illustrated in figure 1 were fabricated and a weighed nickel specimen placed in the bottom of each crucible. The crucibles were loaded with weighed quantities of sodium hydroxide within a dry box, after which the crucible caps were welded in place. This resulted in crucibles which were sealed except for an opening through the vent tubes. The crucibles were evacuated through these vent tubes while the sodium hydroxide was heated to slightly above its melting point. The purpose of this procedure was to dehydrate the sodium hydroxide within the crucibles. In some cases additions were made to the crucibles within a dry box after this dehydration or "purging" process. Next, the vent tubes of the crucibles were connected to a vacuum system by means of which the dry-box atmosphere remaining in the crucibles was removed and the desired atmosphere introduced. The vent tubes were sealed by welding, and two thermocouples attached to each crucible, one near the base of the crucible and the other at the liquid level. These thermocouples facilitated the maintaining of the crucibles at the desired temperature in the neighborhood of 1500° F while they were heated for a period of 24 hours in a furnace especially designed for this purpose. After the furnace runs, the crucibles were opened and the enclosed sodium hydroxide dissolved in water. Weight changes of the specimens were measured and metallographic studies were made, when necessary, to provide a basis for the evaluation of the effects of the various additives.

Crucibles. - In all the experiments described in this report the crucibles used were fabricated from "A" nickel tubing, the analyses of which are as follows:

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Crucible analysis number	Carbon, percent	Iron, percent	Sulfur, percent	Manganese, percent	Copper, percent	Silicon, percent
6	0.10	0.31	0.005	0.16	0.09	0.051
7	.11	.22	.007	.17	.09	.050
8	.11	.05	.007	.16	.07	.049
10	.10	.08	.006	.17	.08	.049
11	.10	.24	.009	.15	.10	.055
12	.11	.28	.005	.18	.06	.049
13	.09	.09	.007	.16	.07	.051
18	.12	.05	.007	.16	.02	.022

All the components of the crucible, except the vent tube, were cut from nickel tubing of 5/8-inch outside diameter and 1/16-inch wall thickness. The flat pieces (end caps and specimens) were made of punchings from lengths of this same tubing which had been split and flattened. The vent tubes were cut from 1/4-inch-outside-diameter nickel tubing of the same nominal analysis as the other parts of the crucible. The crucibles were assembled by welding, using an inert gas atmosphere. This welding procedure was the familiar heliarc process using a shielding atmosphere of helium to which 12.5 percent hydrogen by volume had been added.

When the crucibles were assembled, the welding of the bottoms to the nickel tubing and of the top caps to the vent tubes was performed first. The nickel surfaces were then cleaned by immersion for 2 minutes in a modified aqua regia cleaning solution, washed with distilled water, and dried with acetone. The composition of the nickel cleaning solution was as follows (ref. 1)

Water, cc	1000
Sulfuric acid (66 Be), cc	1500
Nitric acid (38 Be), cc	2250

This solution was allowed to cool and the following was added:

Sodium chloride, g	30
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A weighed specimen, consisting of a coin-shaped disk of nickel 1/16 inch thick and 5/16 inch in diameter, was placed in the bottom of each crucible. Weighed quantities (13.0 ± 0.1 g) of sodium hydroxide pellets of the following analysis (Eimer & Amend, lot 502875) were loaded in the crucibles within a dry box:

NaOH	97.6
Chloride	0.005
Iron (Fe)	0.001
Other heavy metals (as Ag)	0
Carbonate (Na_2CO_3)	0.32
Phosphate (PO_4)	0
Silica and NH_4OH ppt	0
Total nitrogen (as NH_3 , NO_2)	0.001
Sulfate (SO_4)	0

Weighing the sodium hydroxide provided the close control over the liquid level which was found to be necessary for reproducible results.

The crucible tops were welded in place by the modified heliarc process to give the assembled crucible illustrated in figure 1. The crucibles were next subjected to a purging process, after which the desired additions (if any) and atmosphere were introduced in the crucible, and the vent tubes were sealed by crimping and welding. In order to provide a measure of the temperature distribution throughout the crucible, two thermocouples were attached to each crucible by spot-welding at points $1/8$ inch and $2\frac{1}{2}$ inches from the bottom of the crucible. These positions were determined by measurement to give the outside wall temperatures at, or very close to, the bottom of the crucible and the upper liquid level of the molten sodium hydroxide.

Purging system and purging procedure. - The apparatus used in the purging process was a vacuum system and a Variac-controlled furnace as illustrated diagrammatically in figure 2. It consisted of a manifold evacuated by a two-stage mercury diffusion pump and a mechanical fore-pump. A cold trap, cooled with liquid nitrogen, was placed between the vacuum manifold and the mercury diffusion pump to prevent condensable vapors from reaching the latter. The vacuum manifold was equipped with a thermocouple gage reading from 1000 to 1 microns.

In purging, the crucible underwent the following heating and evacuation schedule after connection to the vacuum system: Simultaneous heating and evacuation of the crucibles was begun, with a heating rate such that the crucible temperature increased from room temperature to 400°F over a period of 4 hours. This temperature of 400°F was maintained for 16 hours, after which the crucible temperature was raised to 750°F over a 2-hour period, and maintained at this level until a pressure of 5 microns or less was indicated by the thermocouple gage. About 4 hours of heating at 750°F usually was required.

After this low pressure had been reached in the purging process, the crucibles were allowed to cool and the desired atmosphere, usually

helium at 2 pounds per square inch, was introduced in the vacuum system and hence in the crucibles. When additives were to be tested, the crucibles were transferred to the dry box and weighed quantities of the desired additives were inserted through the vent tubes. The crucibles were reconnected to the vacuum system and again filled with the desired atmosphere. The vent tubes were closed by crimping them, followed by welding, giving the hermetic seal shown in figure 1. The crucibles, after having the thermocouples attached as described, were mounted in the fixture shown in figure 3 for subsequent insertion in the furnace.

Crucible furnace. - Preliminary experience in corrosion studies indicated that the results obtained in one test were very difficult to reproduce in subsequent tests. Consideration of experimental conditions pointed to the possibility of nonuniform temperature distribution throughout the commercially available furnaces used in the preliminary tests. Probing of the furnaces with thermocouples showed that considerable variation in temperature from one part of the furnace to another did exist and that, in general, two different crucibles in the same furnace would not be subject to the same temperature conditions. The need for a furnace which would hold several crucibles simultaneously at the same conditions of temperature (within narrow limits) led to the design and building of a furnace which fulfilled these conditions.

The details of this specially designed furnace are illustrated in figure 4. The bottoms of the crucibles receive heat directly from the nickel plate which separates them from the Globar heating elements, and accordingly are the hottest part of the crucibles. Measurement of the crucible wall temperatures by means of the thermocouples already described indicated that when the crucibles were held at temperatures in the neighborhood of 1500°F , a difference of $45^{\circ}\pm 5^{\circ}\text{F}$ existed in the crucible wall temperatures between a point $1/8$ inch from the bottom of the crucible and a point corresponding to the liquid level, $2\frac{1}{2}$ inches from the bottom. Simultaneous measurements of the temperatures at corresponding points on several crucibles within the furnace showed temperature differences of the order of 10°F .

Four crucibles at a time were loaded in the holder previously described and were inserted in the furnace which already had been brought to operating temperature. The tests were each of 24 hours duration. At the end of this period the holder and the crucibles were removed and allowed to cool rapidly in the air.

Evaluation of results. - After cooling, the crucibles were opened with a tubing cutter and the contents were examined. Following a preliminary observation of the crucible contents, the sodium hydroxide was dissolved with streams of water. The specimen in the bottom of each crucible was recovered, dried, and weighed to obtain a quantitative

measure of the effect of sodium hydroxide without and with various additives. Metallographic studies were made, also, whenever it appeared that any penetration or unusual corrosion had taken place.

When the specimen weight loss was large, a marked effect of the sodium hydroxide on the nickel was evident and further inspection procedures were not applied. In case the weight change was small or the weight actually increased, the specimen was subjected to metallographic studies to determine whether deposition or intergranular penetration occurred.

A further point to consider is the placement of the specimen used in the weight change determinations, that is, whether this specimen occupies a position in the hot or cold zones of the sodium hydroxide melt. It has been observed that the removal of the metal always occurs in the hot zone, leaving a smoothly polished surface. Measurements of this loss are definite and easy to make. Furthermore, the process of solution which leads to this metal removal is fundamental to the processes of both mass transfer and corrosion, since the metal must go into solution before it can transfer. A specimen placed in the cold zone almost invariably shows a weight gain due to the deposition of metal as the end result of mass transfer. There is a possibility that the measurement of weight gains due to these deposits would be less accurate than the measurement of the weight losses in the hot section since several factors may be involved in the deposition of the metal. Prominent among these factors is the possibility that under some conditions the mass-transfer deposit would be poorly adherent and consequently only a fraction of the mass transfer would be measured. Such considerations as these pointed to the measurements of weight losses in the hot zone as being the more desirable.

Experiments with hydrogen atmospheres. - In order to maintain a hydrogen atmosphere, crucibles of the type illustrated in figure 5 were constructed to fit in the furnace used in the other tests described in this report. These crucibles were joined in pairs by means of connecting tubing, allowing a stream of gas to be circulated through both crucibles of the pair, while an air atmosphere was maintained externally. In all other details such as specimens, loading, purging, and thermocoupling, the procedure and conditions were as outlined previously for the single crucibles.

The hydrogen gas used in these crucibles was purified by being passed in turn through a Deoxo unit, drying towers filled with Drierite and a cold trap cooled with liquid nitrogen. The flow of hydrogen was maintained at a slow bubbling rate with the hydrogen discharging into a well ventilated hood after leaving the crucibles. With each pair of hydrogen-filled crucibles, a corresponding set of crucibles containing

helium under static conditions was included in the furnace to provide a basis of comparison for judging the effect of the hydrogen atmosphere.

EXPERIMENTAL RESULTS

Reproducibility

Before the evaluation of the effects of additives on mass transfer, a series of runs was made to establish the degree of reproducibility which could be attained under the conditions described previously. Table I lists the experimental conditions prevailing during, and the results of, three such runs involving four crucibles each. It will be observed that the maximum specimen weight loss was 9.5 milligrams, the minimum weight loss was 7.0 milligrams, giving an arithmetic mean of 7.9 milligrams weight loss for the twelve specimens.

A further statistical evaluation which may be applied to these data is the finding of the average error of a single observation, which is given by the expression

$$a = \pm \frac{\sum d}{n}$$

where $\sum d$ denotes the sum of all the deviations, regardless of sign, and n is the number of observations. The average error of a single observation is seen to be ± 6.9 percent.

Effect of Purging

Justification of the use of the elaborate and time-consuming purging process is provided by the results of two runs listed in table II. It will be noted that in every case the specimens in the unpurged crucibles showed larger weight losses than did the specimens in the corresponding purged crucibles. Furthermore, the unpurged crucibles exhibited evidence of a high internal pressure which resulted in bulging of the vent tubes in all cases and actual bursting of the vent tube welds in two cases. This internal pressure could have been due to the moisture in the unpurged sodium hydroxide, or to gaseous products resulting from reactions involving the moisture.

Additives Having a Detrimental Effect

The following additives proved to have an injurious effect as indicated by increased loss of weight of the specimens or by intergranular penetration of the nickel by sodium hydroxide NaOH. The

experimental conditions under which these additives were tested and the specimen weight changes resulting are given by table III.

Sodium carbonate. - The effect of sodium carbonate Na_2CO_3 is of interest since it is an impurity almost invariably associated with NaOH . The NaOH used in these experiments had a Na_2CO_3 content of 0.32 percent. Additions of 1, 3, and 5 percent Na_2CO_3 in addition to the 0.32 percent already present resulted in the somewhat increased specimen weight losses indicated in table III. It would be of interest and value to compare these results with the effect of carbonate-free NaOH , but such material unfortunately is not available at the time of this writing.

Palladium. - Palladium, added in the form of metal powder, caused a marked increase in the weight loss of the specimens. Examination of the material remaining after the melt was washed away at the end of a run showed that metallic nickel had deposited on the palladium, resulting in a magnetic metallic powder. It is believed that the palladium powder, because of its high density, sank to the bottom and hence occupied the hotter portion of the crucible. The nickel was deposited on the palladium in spite of the fact that it was in the hotter region of the crucible. In the light of previous experiences which invariably had shown the deposition of metallic nickel at the colder regions this formation of nickel deposits in a hot region had not been expected.

Nickel oxide. - One of the hypothetically possible products of reactions between nickel and NaOH is NiO . Assuming for the moment that NiO , or the compound which may be formed when NiO is dissolved in molten NaOH , is an intermediate in the mass-transfer process, it would seem reasonable to suppose that increasing the concentration of NiO or of the resulting compound would tend to suppress the dissolving of nickel in the hot zone. The result actually observed was a slight increase in the weight loss of the specimens, contrary to the speculations just outlined. An explanation of this effect will have to await an elucidation of the mechanism of the mass-transfer process.

Sodium chromate. - Dilute solutions of chromate are commonly used in the protection of ferrous alloys against corrosion by aqueous solutions. It was of interest to determine whether the protective action of the chromate would be exhibited in molten NaOH . Additions of $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$ had the deleterious effects listed in table III.

Chromic sesquioxide. - Additions of anhydrous Cr_2O_3 were made in such quantities that the resulting chromium concentrations were identical with those resulting in the case of the $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$

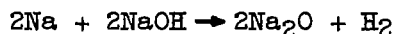
additions. Table III shows that in duplicate runs the largest Cr_2O_3 addition apparently had a beneficial effect in preventing weight loss of the specimens and actually resulted in slight weight gains. Close examination of these specimens, however, revealed a coated appearance. Metallographic studies showed the intergranular penetration illustrated by the photomicrograph in figure 6. This proved to be one instance in which the observation of weight changes alone would lead to an incorrect conclusion regarding the effect of an additive, and pointed out the need for other simultaneous observations (e.g., metallographic studies). Of all the additives tested, Cr_2O_3 was the only one which produced intergranular penetration of nickel. No other additives effected the clear-cut removal of the surface nickel shown in figure 7.

Sodium chlorate. - It has been reported (ref. 2) that the presence of NaClO_3 reduces the corrosive action of molten NaOH on cast iron. The results listed in table III show that such is not the case for nickel under the experimental conditions described here; instead, the presence of NaClO_3 is definitely harmful.

Sodium oxide. - Additions of Na_2O had pronounced effects in increasing both the deposition of nickel crystals in the colder portion of the crucible and the weight losses of the specimens (table III). In fact, Na_2O proved to be one of the more deleterious of all the additives tested.

Sodium hydride and lithium hydride. - In view of the fact that the oxidizing agents which were tested as additives had detrimental effects, it seemed possible that reducing agents might have opposite effects. Two powerful reducing agents, NaH and LiH , proved to be extremely detrimental, as shown by the results in table III.

Unpublished data by Desmon and Mosher, of this laboratory, show that additions of metallic sodium to NaOH increase the mass transfer of nickel in a similar manner. As shown previously, the presence of Na_2O greatly accelerates the mass transfer of nickel in NaOH . Metallic sodium reacts as follows with molten NaOH :



Therefore, additions of metallic sodium are equivalent to additions of Na_2O , and it is not surprising that both have the same effect. It is considered likely that NaH and LiH undergo similar reactions with NaOH , also resulting in the formation of Na_2O and Li_2O . This could account for the similar effects of additions of Na_2O , Na , NaH , and LiH in accelerating mass transfer.

Additives Having No Marked Effects

Sodium chloride. - The effect of NaCl was of interest in connection with the additions of NaClO₃ previously described, since at the temperatures of these experiments NaClO₃ is decomposed to form NaCl and O₂. Additions of NaCl in the quantities which would be formed by the decomposition of the NaClO₃ additions showed no observable effects (table IV). Apparently NaCl is completely inert as an additive, and the deleterious effects of NaClO₃ may be attributed to the presence of the evolved oxygen.

Calcium nitride. - Because additions of calcium nitride Ca₃N₂ were without pronounced effects (table IV), Ca₃N₂ is considered to be inert when used as an additive.

Additives Having Beneficial Effects

Sodium aluminate. - Relatively large additions of NaAlO₂ have been found successful in practically eliminating mass-transfer effects under static conditions (ref. 3). Results listed in table V appear to substantiate this report insofar as weight changes are concerned. Examination of the inner walls of the crucibles showed decreased deposition of nickel.

A subsequent report (ref. 4) stated that the solubility of NaAlO₂ in molten NaOH is relatively low (of the order of 1 percent) and raised the speculation that the beneficial action of the NaAlO₂ additions was due to "blanketing" of the specimens in the bottom of the crucibles by the undissolved additive. Dynamic tests are recommended for determining whether this is the case, and whether NaAlO₂ additions are of value in systems with high fluid velocities.

Calcium hydride. - Additions of CaH₂ were moderately beneficial in decreasing the weight losses of the specimens (table V). It will be noted that the beneficial effect showed little tendency to vary with the quantity of CaH₂ added.

Hydrogen. - The presence of hydrogen also has been reported (ref. 5) to overcome corrosion and mass-transfer effects. Experiments carried out with a slow stream of hydrogen flowing through crucibles of the type illustrated in figure 5 showed that hydrogen was effective in reducing the weight changes of the specimens to a very low value (table V). There was still evidence of mass transfer, however, in the form of a slight deposit of nickel crystals at the uppermost level of the molten caustic.

SUMMARY OF RESULTS

The effect of a number of additives on the corrosion and mass-transfer behavior of nickel in the presence of molten sodium hydroxide in the region of 1500° F was investigated in static crucibles. It was found that close attention to experimental conditions, particularly the temperature distribution, the liquid level within the crucible, and the condition of dehydration, was necessary to obtain a degree of reproducibility which would insure confidence in the results.

The investigation indicated that:

1. A number of additives (anhydrous sodium carbonate, palladium, nickel oxide, hydrated sodium chromate, chromium oxide, sodium chlorate, sodium oxide, sodium hydride, and lithium hydride) were detrimental in that they increased the weight losses of the specimens. In addition, the specimens from the crucibles to which chromium oxide had been added showed appreciable intergranular penetration.
2. A few additives (sodium chloride and calcium nitride) appeared to be without effect on weight change of specimens by sodium hydroxide.
3. Additives that reduced the weight loss caused by sodium hydroxide were: calcium hydride, sodium aluminate, and hydrogen.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, April 29, 1953

REFERENCES

1. Anon.: Metals Handbook, 1948 Edition. Am. Soc. Metals (Cleveland), 1948, p. 1041.
2. Perret, Ugo: Corrosion of Pots Used in the Fusion of Caustic Soda. *Chimica e Industria* (Italy), vol. 20, 1938, pp. 133-136. (Abs. from Chem. Abs., vol. 32, no. 20, 1938, p. 7880.)
3. Craighead, C. M., Smith, L. A., Phillips, E. C., and Jaffee, R. I.: Continued Studies of Corrosion by Fused Caustic. Rep. No. BMI-794, Metallurgy and Ceramics, Battelle Memorial Inst., Dec. 18, 1952. (Contract No. W-7405-eng-92.)
4. Fitz, R. A., and Stang, J. H.: Thermal-Convection Harp Tests. Progress Report for Nov. 1952. Rep. No. BMI-790, Metallurgy and Ceramics, Battelle Memorial Inst., Dec. 1, 1952, p. 53. (Contracts No. W-7405-eng-92; No. NObS-50352.)

5. Cottrell, W. B., ed.: Aircraft Nuclear Propulsion Project Quarterly Progress Report. Rep. ORNL 1170 for period ending Dec. 10, 1951. Carbide and Carbon Chemicals Co. (Oak Ridge, Tenn). (Contract No. W-7405, eng. 26.)

TABLE I. - MASS-TRANSFER EFFECTS IN CRUCIBLES WITHOUT ADDITIVES,
SHOWING REPRODUCIBILITY OF RESULTS

Experiment number	Crucible number	Crucible analysis number	Temperature, °F		Specimen weight change, g	Deviation from mean, g
			Bottom of crucible	Liquid level		
9	33	6	1527	1475	-87×10^{-4}	7.7×10^{-4}
	34	6	1529	1478	-71	8.3
	35	6	1517	1475	-72	7.3
	36	6	1527	1480	-82	2.7
10	37	6	1539	1497	-95	15.7
	38	6	1541	1493	-79	.3
	39	6	1529	1492	-78	1.3
	40	6	1530	1489	-76	3.3
14	53	6	1541	1495	-86	6.7
	54	6	1536	1488	-70	9.3
	55	6	1538	1494	-76	3.3
	56	5	1546	1497	-79	.3
Mean					-79.3	

TABLE II. - EFFECT OF DEHYDRATION OF SODIUM HYDROXIDE ON
MASS-TRANSFER EFFECTS

Experiment number	Crucible number	Crucible analysis number	Purged	Temperature, °F		Specimen weight change, g
				Bottom of crucible	Liquid level	
31	121	11	No	1539	1491	-406×10^{-4}
	122	11	No	1541	1489	-362
	123	12	No	1528	1487	-187 ^a
	124	7	Yes	1532	1482	-56
32	125	10	Yes	1539	1498	-93
	126	10	No	1544	1495	-294
	127	10	No	1535	1493	-367
	128	10	No	1534	1493	-320

^aPartly purged.

TABLE III. - EFFECTS OF ADDITIVES WHICH INCREASE MASS TRANSFER
OR CORROSION, OR BOTH

Experiment number	Crucible number	Crucible analysis number	Additive	Temperature, °F		Specimen weight change, g
				Bottom of crucible	Liquid level	
18	69	7	None	1539	1495	-70×10^{-4}
	70	7	1 percent Na_2CO_3	1543	1495	-175
	71	7	3 percent Na_2CO_3	1530	1492	-193
	72	7	5 percent Na_2CO_3	1536	1489	-220
23	89	12	None	1539	1495	-69
	90	12	1 percent Na_2CO_3	1544	1497	-185
	91	12	3 percent Na_2CO_3	1531	1492	-175
	92	12	5 percent Na_2CO_3	1531	1490	-197
12	45	6	None	1539	1495	-75
	46	6	2 percent Pd	1548	1500	-606
	47	6	2 percent Pd	1536	1493	-329
	48	6	5 percent Pd	1541	1493	-862
25	97	8	None	1539	1485	-104
	98	8	0.39 percent NiO	1543	1489	-173
	99	8	1.2 percent NiO	1530	1489	-142
	100	8	1.9 percent NiO	1539	1487	-184
29	113	11	None	1539	1501	-71
	114	11	0.39 percent NiO	1552	1504	-86
	115	11	1.2 percent NiO	1536	1495	-123
	116	11	1.9 percent NiO	1542	1494	-158
20	77	12	None	1539	1492	-58
	78	12	1 percent $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$	1545	1500	-211
	79	12	3 percent $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$	1539	1492	-195
	80	12	5 percent $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$	1539	1492	-141
27	105	11	None	1539	1497	-62
	106	11	1 percent $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$	1549	1501	-196
	107	11	3 percent $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$	1539	1497	-224
	108	11	5 percent $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$	1547	1498	-205

TABLE III. - Concluded. EFFECTS OF ADDITIVES WHICH INCREASE MASS
TRANSFER OR CORROSION, OR BOTH

Experiment number	Crucible number	Crucible analysis number	Additive	Temperature, °F		Specimen weight change, g
				Bottom of crucible	Liquid level	
24 ^a	93	12	None	1539	1499	-89x10 ⁻⁴
	94	12	0.18 percent Cr ₂ O ₃	1543	1499	-118
	95	12	0.53 percent Cr ₂ O ₃	1534	1493	-79
	96	8	0.89 percent Cr ₂ O ₃	1534	1493	22 ^a
28	109	11	None	1539	1495	-69
	110	11	0.18 percent Cr ₂ O ₃	1546	1497	-121
	111	11	0.53 percent Cr ₂ O ₃	1536	1492	-114
	112	11	0.89 percent Cr ₂ O ₃	1540	1491	2 ^a
17	65	7	None	1539	1492	-79
	66	7	1 percent NaClO ₃	1544	1495	-222
	67	7	3 percent NaClO ₃	1536	1493	-286
	68	7	5 percent NaClO ₃	1531	1488	Leaked
22	85	12	None	1539	1497	-59
	86	12	1 percent NaClO ₃	1548	1500	-214
	87	12	3 percent NaClO ₃	1539	1496	Leaked
	88	12	5 percent NaClO ₃	1542	1497	Leaked
33	129	10	None	1539	1493	-129
	130	10	0.1 g NaH	1543	1495	-264
	132	10	0.2 g NaH	1536	1491	-678
	131	10	0.5 g NaH	1533	1487	-1627
37	145	13	None	1539	1491	-60
	146	13	0.1 g LiH	1544	1494	-665
	147	13	0.3 g LiH	1535	1489	Leaked
	148	13	1.0 g LiH	----	----	Exploded
49	169	18	None	1539	1497	-44
	170	18	1 percent Na ₂ O	1534	1494	-132
	171	18	3 percent Na ₂ O	1532	1494	-398
	172	18	5 percent Na ₂ O	1532	1489	-766

^aSpecimens showed intergranular penetration.

TABLE IV. - ADDITIVES HAVING NO APPRECIABLE EFFECTS

Experiment number	Crucible number	Crucible analysis number	Additive	Temperature, °F		Specimen weight change, g
				Bottom of crucible	Liquid level	
30	117	11	None	1539	1494	-69×10^{-4}
	118	11	0.55 percent NaCl	1546	1494	-65
	119	11	1.7 percent NaCl	1539	1487	-72
	120	11	2.8 percent NaCl	1539	1488	-66
35	138	10	None	1562	1513	-167
	139	10	0.2 g Ca_3N_2	1550	1512	-250
	140	10	1.0 g Ca_3N_2	1552	1509	Leaked
39	153	13	None	----	----	-100
	154	13	0.2 g Ca_3N_2	1615	----	-96
	155	13	1.0 g Ca_3N_2	1607	1564	-121

TABLE V. - EFFECTS OF ADDITIVES BENEFICIAL IN
REDUCING MASS TRANSFER

Experiment number	Crucible number	Crucible analysis number	Additive	Temperature, °F		Specimen weight change, g
				Bottom of crucible	Liquid level	
19	73	7	None	1539	1492	-56×10^{-4}
	74	7	5 percent NaAlO_2	1561	1492	-4
	75	12	10 percent NaAlO_2	1558	1487	-4
	76	12	15 percent NaAlO_2	1556	1480	0
26	101	11	None	1539	1495	-78
	102	11	5 percent NaAlO_2	1560	1494	-53
	103	11	10 percent NaAlO_2	1533	1500	13
	104	11	15 percent NaAlO_2	1551	1487	-16
34	133	10	None	1541	1496	-185
	134	10	0.1 g CaH_2	1546	1498	-74
	136	10	0.2 g CaH_2	1534	1489	-68
	137	10	1.0 g CaH_2	1538	1487	-77
38	149	13	None	1550	1501	-160
	150	13	0.1 g CaH_2	1529	1494	-69
	151	13	0.2 g CaH_2	1533	1493	-81
	152	13	1.0 g CaH_2	1541	1497	-61
36	141	10	H_2	1536	1493	+5
	142	10	H_2	1562	1495	-6
	143	10	None	1522	1476	-67
	144	10	None	1520	1476	-58
42	165	17	None	1539	-----	-58
	166	17	None	1550	-----	-78
	167	17	H_2	1549	1491	-14
	168	17	H_2	1532	1484	-4

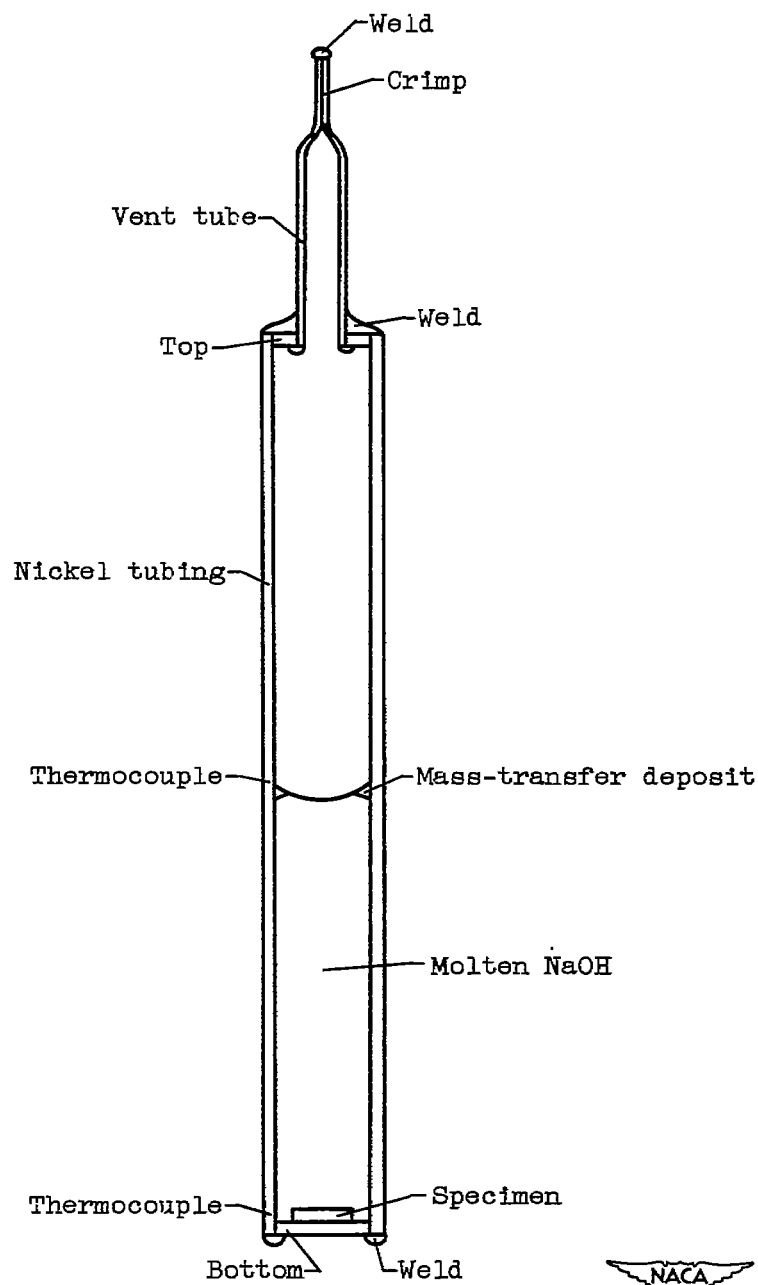


Figure 1. - Drawing of static capsule.

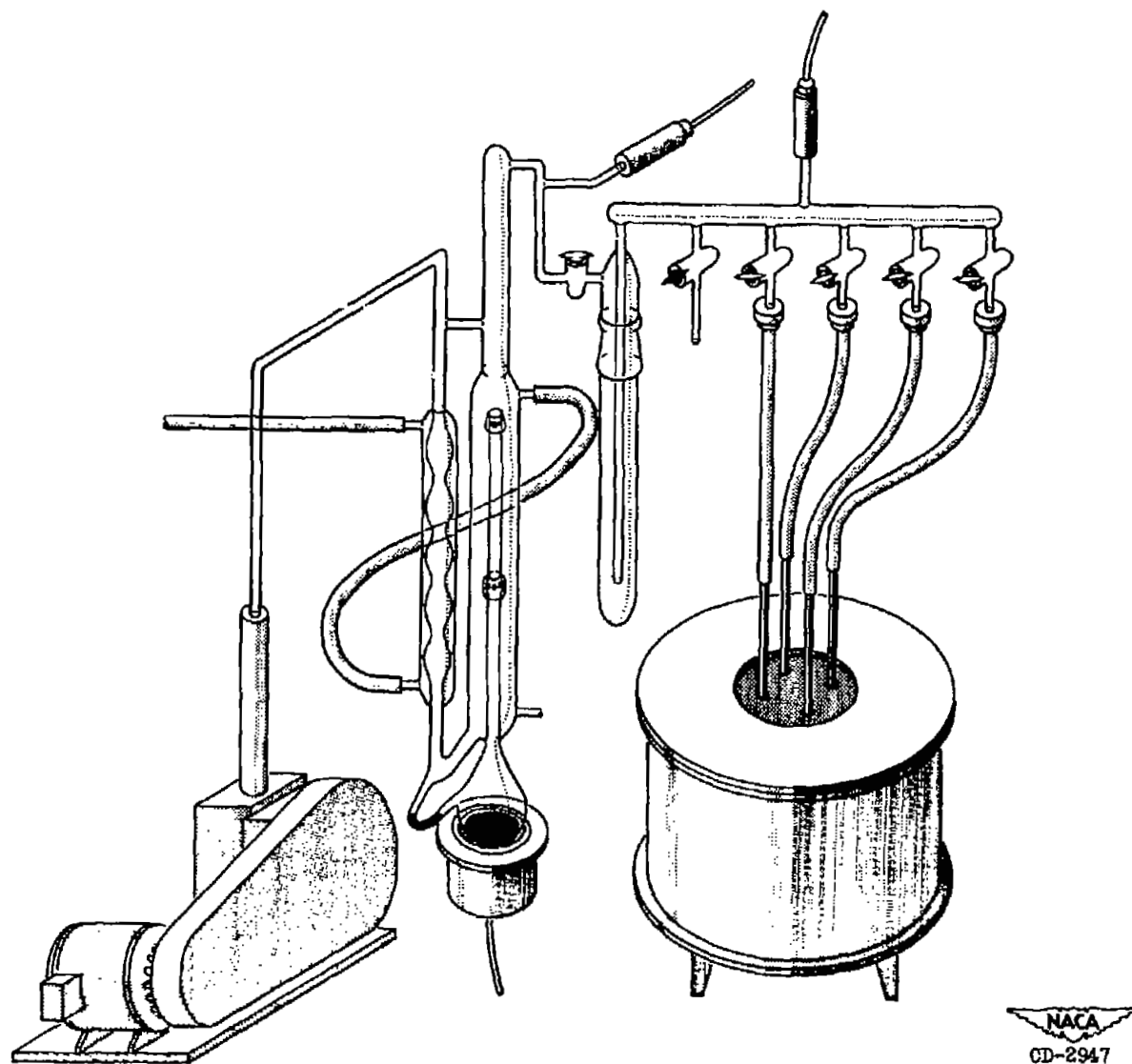


Figure 2. - Apparatus used for dehydration of caustic.

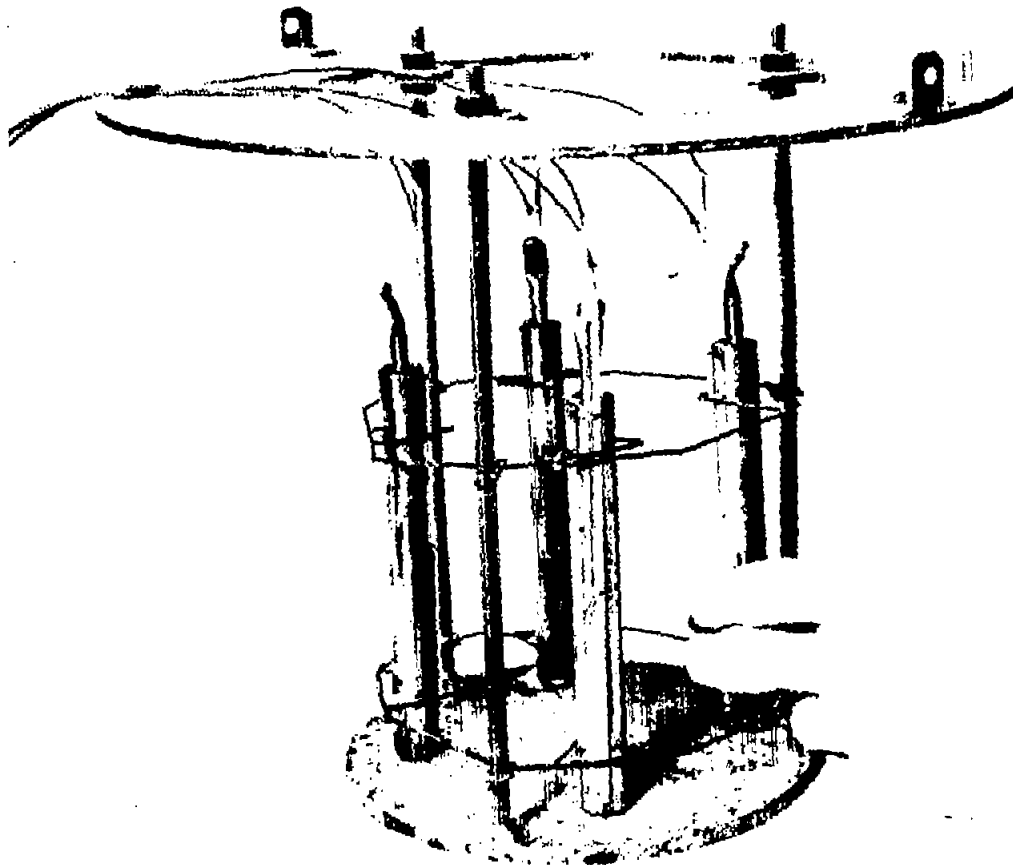


Figure 3. - Photograph of crucible holder with crucibles in position.



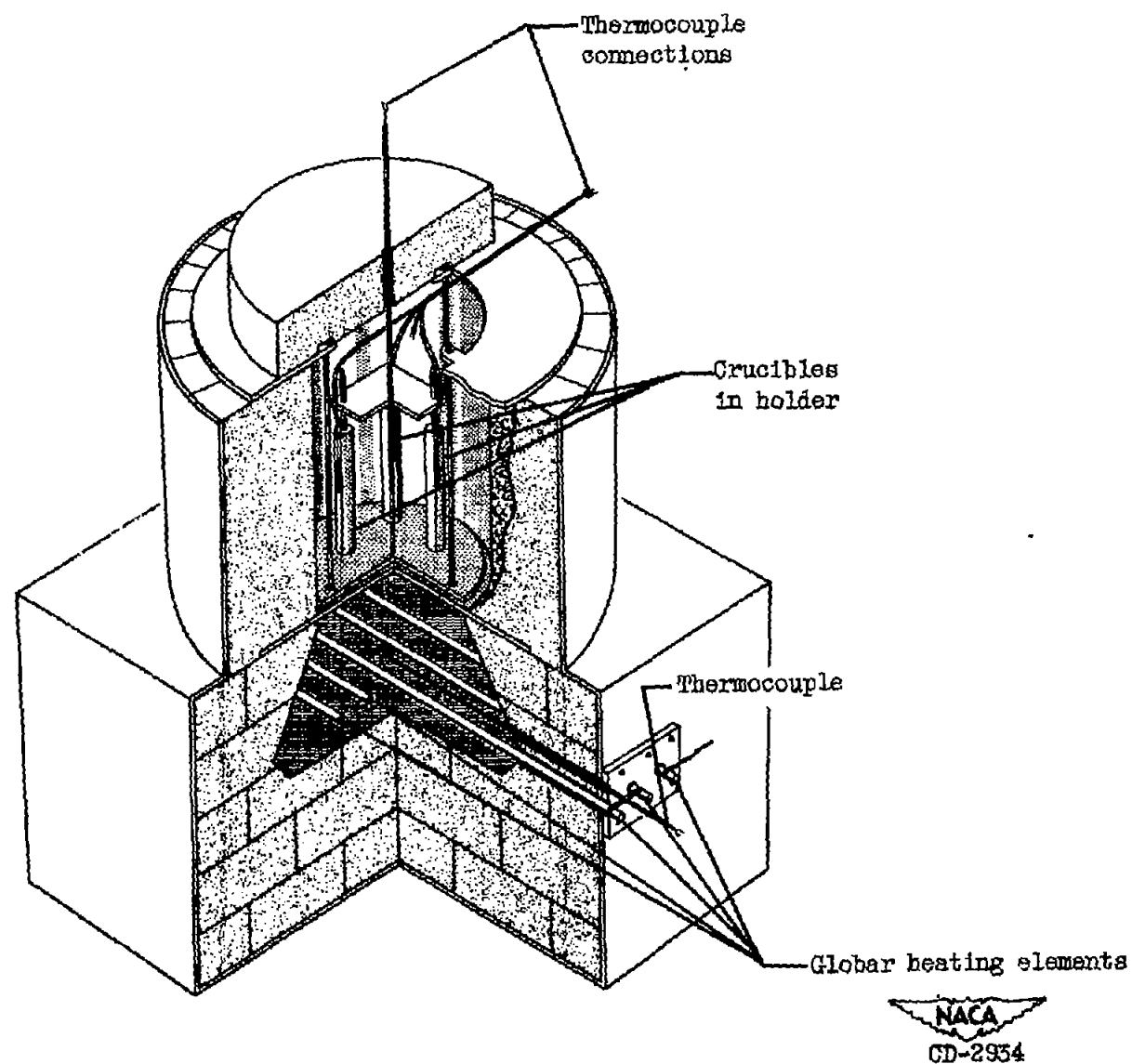
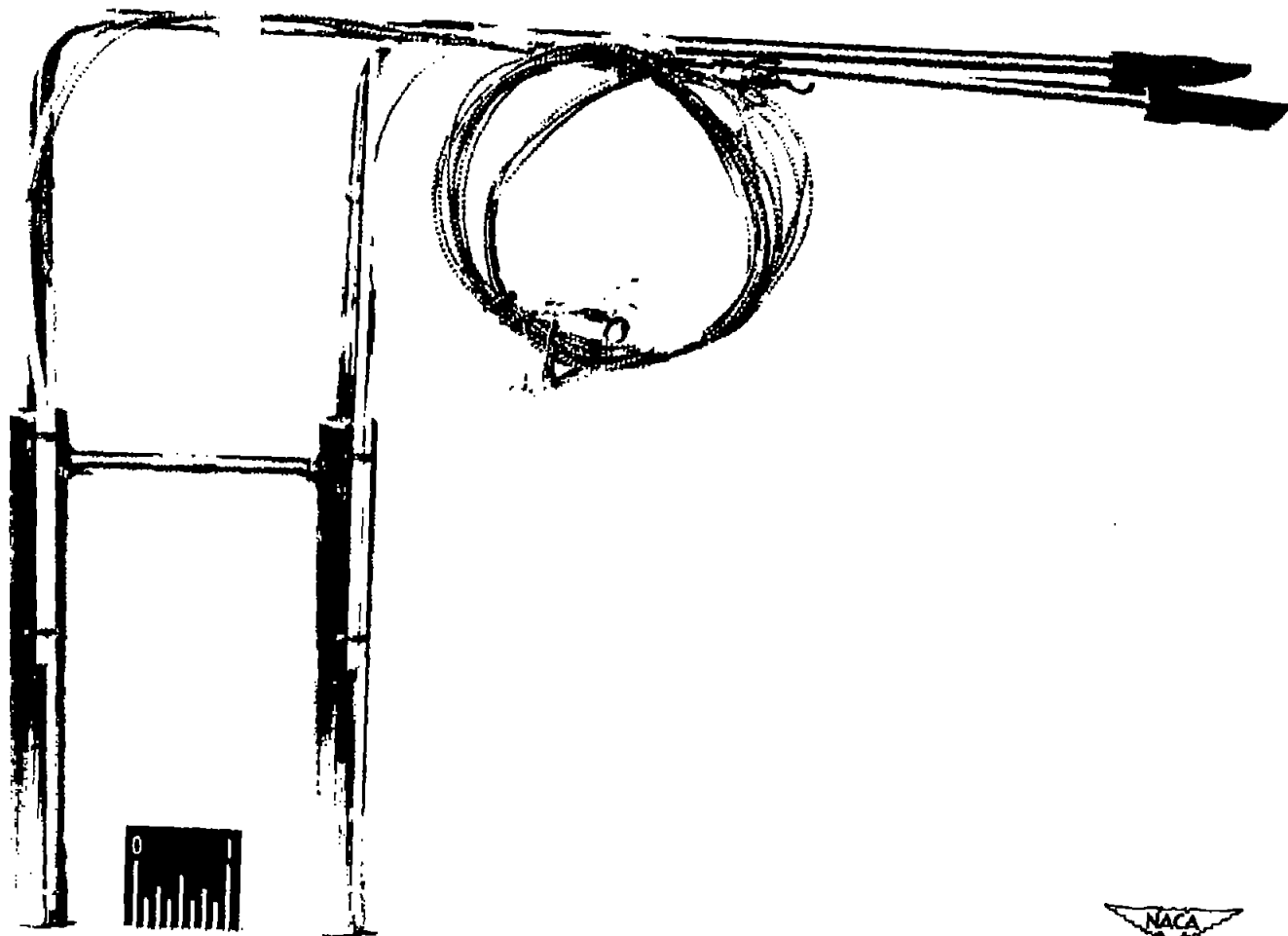
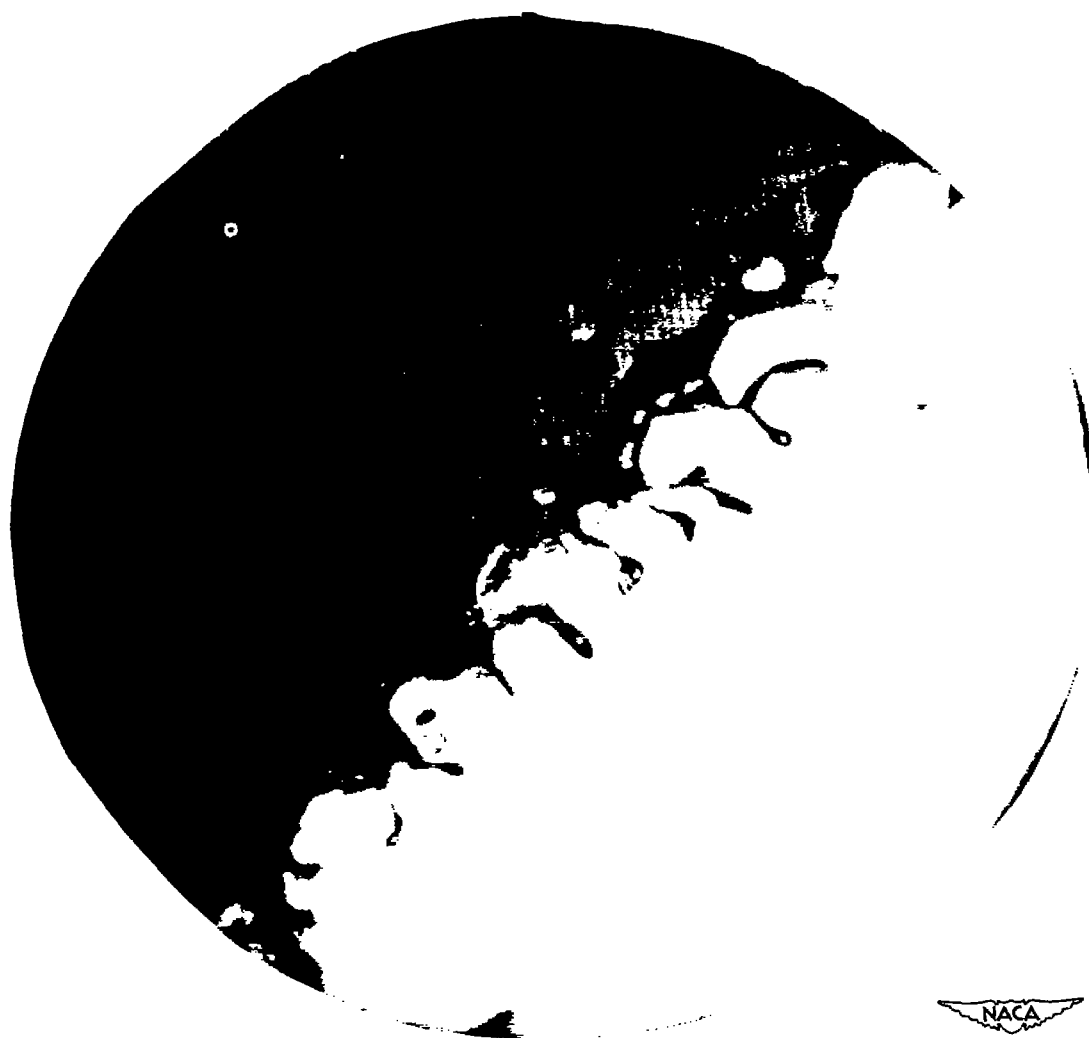


Figure 4. - Diagram of crucible furnace.



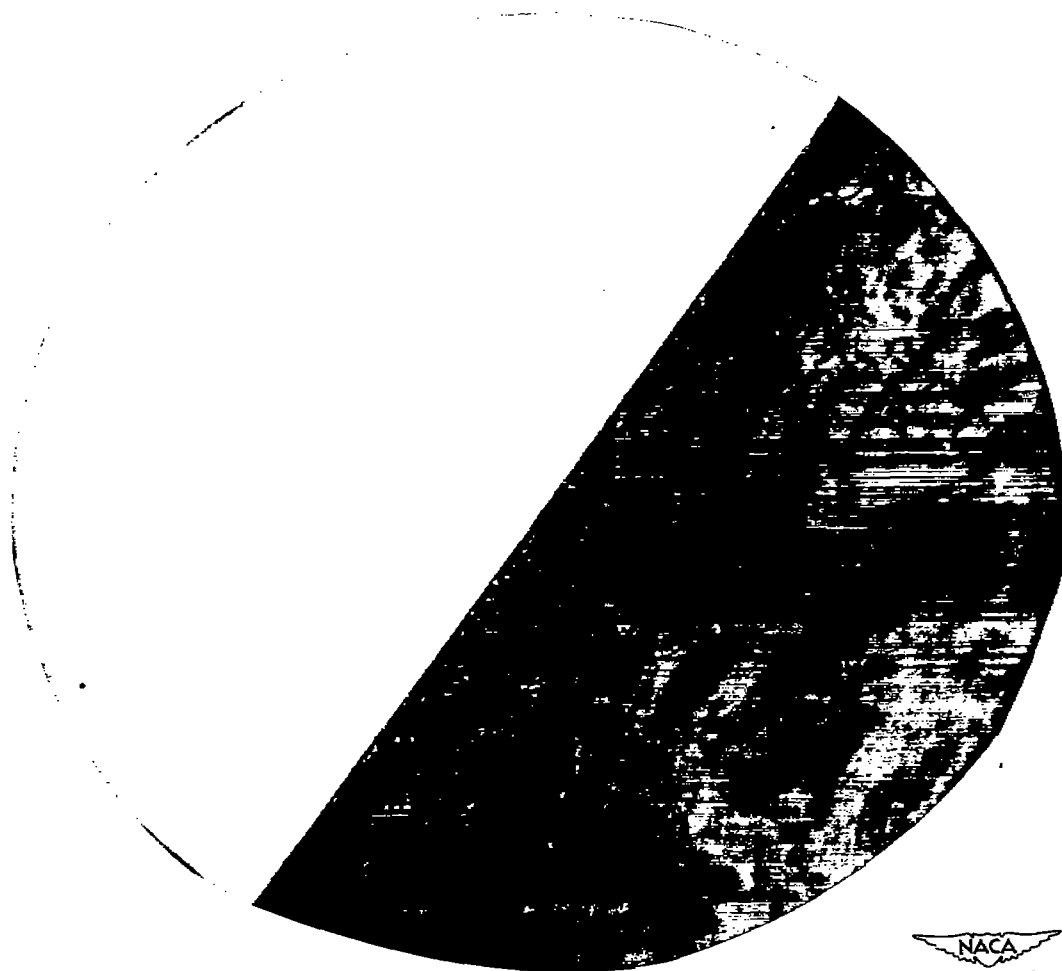
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Figure 5. - Crucibles arranged for circulating hydrogen atmosphere.



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Figure 6. - Photomicrograph showing pronounced intergranular penetration of nickel specimen exposed to sodium hydroxide and chromium sesquioxide.



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Figure 7. - Photomicrograph of edge of nickel specimen after exposure to sodium hydroxide, showing absence of intergranular penetration.

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